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This report provides an overview of the work performed during the Phase I, where the growth of Al_2O_3 on GaAs by gas source molecular beam epitaxy was demonstrated. We start with a description of the system that was constructed, and present results of the Al_2O_3 on GaAs thin film growth.

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Final Report

GaAs - Based MOSFET Employing Epitaxial Al_2O_3

Phase I

June 1998

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This report provides an overview of the work performed during the Phase I, where the growth of Al_2O_3 on GaAs by gas source molecular beam epitaxy was demonstrated. We start with a description of the system that was constructed, and present results of the Al_2O_3 on GaAs thin film growth.

Growth Facility

The system GEM Research Inc. established during Phase I for the growth of the Al_2O_3/GaAs heterostructures, consists of a gas source molecular beam epitaxy (GS-MBE) facility. Al_2O_3 is grown employing conventional Al effusion cell and N_2O gas source.

The solid Al and As sources (manufactured by Varian) are connected to a Eurotherm FICS 10 control module equipped with a power supply and temperature controller. Gas inlets are welded through a 6" flange and are valved off immediately outside the vacuum chamber to minimize pumping volume. Stainless steel, quarter-inch pipes are guided through mass flow controllers calibrated for nitrous oxide and hydrogen at 100 sccm and 200 sccm, respectively. The pipes are connected to a gas cabinet. The inside of the cabinet was redesigned to accommodate the purge line, connected from the outside, and the fittings for the N_2O and H_2 gas tanks. The non-hazardous nitrogen tank providing the purge gas is placed outside the cabinet. A compressed air tank provides the 90 PSI pressure necessary to operate the pneumatic valve between the growth chamber and the turbomolecular pump. The cabinet contains a high purity nitrous oxide tank. The mass flow controllers are operated from the four-channel MFC Power Supply and Readout produced by MKS.

Pumping of the chamber is provided by the built-in ion pump and attached 500 l/s turbomolecular pump. The ion pump is employed during its idle periods and is closed off during growth, since pressures then exceed 10^{-4} torr. The turbo pump is vented to the outside. Base pressures during idle are 10^{-8} torr after opening and lower by an order of magnitude after baking and cryocooling.

The GaAs substrates are mounted on a 2 inch substrate holder with a 15 amp heater. With power from a HP 60 V, 15 A, DC power supply the substrate can be raised to 1100°C if required.

Substrate temperature was determined by a pyrometer capable of reading temperatures above 350°C to within 10°C.

During Phase I the substrate could not be rotated and therefore RHEED characterization has been limited to a single azimuth per growth run. However, we intend to modify the sample holder to enable rotation.

Growth

As described above the growth chamber was equipped with gas inlets for nitrous oxide and Knudsen cells containing solid Al and As. The Al cell generated the following fluxes at the respective cell temperatures:

T (°C)	Flux (torr)
828	1.3×10^{-8}
1000	2.4×10^{-8}
1097	4.0×10^{-8}
1228	2.0×10^{-7}

The growth rate at these fluxes was obtained for the sticking coefficient 1 of Al (100% of the atoms arriving at the growth surface stayed on the surface). For a flux of 2.0×10^{-7} torr that was 0.45 μm per hour. However, for the substrate temperatures greater than 650°C the sticking coefficient is no longer equal to 1 and it decreases with increasing temperature. This issue will be addressed in the growth and characterization section below.

The gas flow rate was maintained and monitored by flow controllers which were set to deliver 40 sccm of N_2O. The base pressure in the chamber was approximately 5×10^{-7} torr and could be brought down to 1×10^{-7} torr with liquid nitrogen cooling. Cooling as well as changing of chamber volume by closing off the ion pump were utilized to control the pressure during growth. Chamber pressure during the deposition (when the gases were delivered) varied between 3×10^{-3} and 2×10^{-2} torr.

The oxide desorption and the growth were monitored by reflection high energy electron diffraction (RHEED).

Growth of Al_2O3 was performed at 650, 700, 750 and 800. As mentioned above the growth rate varied with temperature. It was 280Å per hour at 750°C. The thickness of the layers was measured by ellipsometry, alpha step and RBS. During these investigations the gas flow and Al flux were kept constant with variation in the substrate temperature only.

Due to the low quality of the aluminum oxide layers grown directly on GaAs (100) we have decided to deposit an AlAs buffer between GaAs and Al_2O_3. The rationale behind this new strategy is intimately associated with the temperature gap between the maximum growth temperature of GaAs (680°C) and the lowest growth temperature of single crystal γ-Al_2O_3 (750°C).

An attempt to grow aluminum oxide directly on gallium arsenide leads to low quality, polycrystalline Al_2O_3 with interface defects, which is undesirable from the standpoint of its potential application as a gate dielectric. However, gallium arsenide decomposes at temperatures higher than approximately 680°C, and therefore, there exists very little freedom in varying the growth conditions of the oxide.

Utilizing AlAs allows us to fill the temperature gap since this compound can be grown in the range of temperatures between 580 and 750°C. It may also reduce density of interface defects and their impact on the gate field in the GaAs channel due to the lattice match between AlAs and GaAs. We have managed to grow AlAs films of thickness of several hundred nanometers and then reduced it to tens of nanometers. The aluminum oxide overgrowth performed at 780°C shows a mirror like surface and stochiometric composition on the thicker films.

We have found some As incorporation into Al_2O_3. The origin of the As presence may stem from either interdiffusion or background arsenic remaining from the AlAs growth. We have introduced a time delay between the AlAs and Al_2O_3 growth in order to lower the temperature of the As cell. We have also reduced the growth temperature from 800 to 750°C to minimize any interdiffusion. Finally, we initiated the aluminum oxide deposition with approximately two monolayers of Al before allowing oxygen into the chamber.

Spinel Structure

When the growth is performed by MBE in a temperature range between 750°C and 900°C the aluminum oxide turns out be in a γ phase, which has cubic symmetry. A γ phase of aluminum oxide is a form of spinel structure (Al_2MgO_4) with oxygen atoms in an almost perfect cubic close-packing arrangement with the metal atoms lying in holes of the packing. In this crystal each magnesium atom is tetrahedrally surrounded by oxygens and each aluminum by six octahedrally distributed oxygens (a_0 = 8.0800 A). Spinels form solid solutions with simpler oxides such as Al_2O_3, however, the differences in the chemical formulas of the compound entering into the solution make the crystal structure incomplete, with deficiencies in the oxygen close-packing or because of incomplete filling of the possible metal positions. γ-Al_2O_3 is an extreme example of a deficit structure (a_0 = 7.90 A) with vacancies in oxygen and aluminum positions. Some preparations of the oxide have deficit arrangements based on hausmannite structure (with a unit cell a_0 = 5.62 A, c_0 = 7.79 A, a_0' = 7.95 A). It is not clear what arrangement layers grown by Dr. Ishida exhibit considering a lack of substantial demonstrative data. One of the important consequences of this type of structure is that it makes the oxide highly adsorptive meaning that there is an energy consideration leading to a need to fill the voids i.e. it is possible that arsenic or other material present is incorporated into the aluminum oxide. It is also unclear how the γ oxide matches up with a diamond surface net and whether they are coherent. γ- Al_2O_3 forms at lower than α- Al_2O_3 temperatures and comprises of variety of phases. These phases represent different degrees of ordering of Al atoms in the deficit structure since there are only 21 and 1/3 metal atoms arranged at random in the 16 octahedral and 8 tetrahedral positions of that structure. The X-ray diffraction and electron diffraction studies show the following transitions:

boehmite --450°C--> γ --750°C--> δ --1000°C --> θ + α --1200°C --> α

bayerite --230°C--> η --850°C --> θ --1200°C --> α

γ– Al_2O_3 has a defect spinel structure in which there is a random arrangement of the metal ions. The δ phase is a tetragonal spinel superstructure with a tripled c-axis in which there are ordered

vacancies in the octahedral sites. The θ phase is isostructural with β-Ga_2O_3. In all three forms the O atoms are c.c.p. or approximately so, and the structural changes involve only movements of cations.

The above information has to be verified for the MBE growth since it differs thermodynamically from the other methods of crystal growth. All the above phases entail different band structures and thus there may be differences in the band offset between the oxide and the gallium arsenide as well as the varying index of refraction. However, since all the phases depend on the conditions of the crystal growth, they are likely to be controllable, thereby adding flexibility to the material.

Results

During growth of the Al_2O_3, the RHEED pattern was (1x1) for the first five minutes of growth and then turned spotty suggesting Stranski-Krastanov mode of growth, which still preserves crystallinity as demonstrated by the XRD data. The surface of the grown samples exhibited a blue tint and was mirror-like, indicating good epitaxial growth.

We have characterized the samples grown both on GaAs and Si for comparison with a scanning electron microscope (SEM), X-ray diffraction and secondary ion mass spectroscopy (SIMS). SEM micrographs show a good surface morphology on the aluminum oxide film grown at 750°C. By comparison, a micrograph of a sample grown at a low temperature (650°C) indicates a rough, grainy surface.

The X-ray diffraction results are consistent with, and indicate the existence of the crystalline form of γ-Al_2O_3. The small peak width suggests small mosaic spread and good quality of the crystalline thin film.

The X-ray diffraction data described above allowed us to look only at the growth direction of the crystal. To obtain more information about the crystal it was necessary to look off the axis, i.e. in the direction other than normal to the sample surface. To achieve this purpose we utilized a General Area Detector Diffraction System, which is based on the Debye-Scherr focusing scheme. When a monochromatic x-ray beam is applied to a randomly oriented sample

diffraction will occur in cones. The opening of each cone is a function of the 2θ angle of the diffracting plane for that cone. Low 2θ angles have narrow cones and high 2θ angles have wide, open cones of diffraction. When a general area detector intersects these cones, it records a series of rings (Debye rings). The data frame collected shows the diffraction rings across the 2-dimensional image with the radius being the 2θ angle and the position on the ring varying with the chi angle. If the sample is definitely oriented the intensity spots will show with well defined 2θ and χ angles. We have observed bright spots, present in all of the analyzed samples, consistent with the (311) and (222) orientation of the γ-Al_2O_3. These spots allowed us to determine the lattice spacing in the (311) and (222) directions and estimate the coherence length in those directions. The in-plane lattice constant appears to be unchanged from the nominal value (7.90 A) suggesting that the crystal is not strained. The coherence length related to the FWHM of the peak (0.25 degrees) indicates good crystalline quality.

The SIMS profile obtained from a structure incorporating AlAs shows all the layers (i.e. GaAs-AlAs-Al_2O_3) with the correct stochiometry. There are, however, signs of As and O interdiffusion at the interface of AlAs and Al_2O_3.

Technological Issues

The epitaxial growth of aluminum oxide has posed quite considerable technical difficulties associated with oxidizing environment at high temperature.

Parts which are particularly sensitive are the sample heater and Al cell, which are at temperatures above 900°C in NO_2. The heaters we have used so far are made out of molybdenum and stainless stainless steel with ceramic isolating parts. Both Mo and steel oxidize at such high temperatures, become very brittle and break down easily. The effusion cell and the crucible also undergo degradation due to the fact that all the metal parts oxidize in a standard cell and the boron nitride of the effusion cell decomposes by losing boron to form boron oxide. We have managed to avoid the breakdown of the cell so far with exception of the thermocouple. However, the thermocouple is not essential due to the fact that the cell heater has been calibrated and all the temperature readings have been related to the power readings (current readings). The degradation

of the crucible is slow and even though its failure is imminent it is possible to utilize it for several months. There exist commercially produced cells able to withstand the corrosive environment described above. They are mostly made from Hayes alloy and employ silicon carbide parts. However, their price is in the range of $20,000 to $30,000 (EPI) which contributes to our decision of delaying the purchase until all other options are exhausted.

We have purchased a heater whose body is made out of alumina and where heating element is a platinum-rhenium wire. The heater permits mounting of two inch wafers. The larger heating area will improve temperature uniformity across a sample, which directly contributes to the layer thickness uniformity. We expect the heater to able to reach approximately 1200°C with the power consumption of about 1000 W. If it is necessary, two existing power supplies will be connected in series to provide sufficient power.

The alternative to the aluminum effusion cell is a tri-methyl-aluminum (TMA) gas source, however, the cost of setting up the gas line and the requisite equipment, as well as the organic material contamination does not warrant, at present time, the installation of this type of equipment.

Due to the above mentioned problems, we have undertaken a major modification of the growth system, installing differentially pumped cells to protect them from the oxygen environment, a thermocouple probe protected by a Ni based shield, and a transfer system enabling quick and efficient exchange of samples without braking vacuum in the main system. All of the components exposed to oxygen at high temperature are made of ceramic or Hestelloy 230, which is resistant to oxidation and nitration.

Future Research

The efforts at the GEM Research laboratory will concentrate on electrical characterization of single layers. A MOS capacitor will be fabricated by depositing aluminum on top of the aluminum oxide layer. The deposition will be performed in a metalization chamber through a mask providing the ability to measure capacitance independently at different points of the sample. The capacitance measurement will utilize both high and low frequency AC voltage to determine the response of the majority and the minority carriers respectively. We will

also measure the maximum electric field the oxide can support before breaking down to determine its potential as a gate oxide.

I-V characteristics of the MOSFET structure will be evaluated and the carrier mobilities of the channel layer will be determined utilizing the Hall effect. The Hall effect will also be used to assess carrier densities in epitaxially grown AlAs layers..